



MECHANOCHEMICAL SOLID STATE REACTION OXIDATION COUPLING OF BETA-NAPHTHOLE TO BINAPHTHOLE WITH $\text{FeCl}_3 \times 6\text{H}_2\text{O}$ USING HIGH SPEED VIBRATION MILL AND INVESTIGATION OF PARAMETERS EFFECTS IT ON THE YIELD AND REACTION TIMES

K. Shayesteh¹, J. Moghddas¹, M. Haghghi¹, G. Imanzadeh²

1 - Sahand University of Technology, Chemical Engineering Department, Tabriz, Iran

2 - Chemistry Group, The University of Mohaghegh Ardabili, Ardabil, Iran

k_shayesteh2000@yahoo.com

Mechanochemical processes (MCP) use mechanical energy to activate substances by developing structural changes. The majority of the review articles dates to the beginning of the written history of mechanochemistry to the end of 19th century. The establishment of mechanochemistry as a separate branch of chemistry is usually attributed to Matthew Carey Lea at the end of the 19th century, who demonstrated that halides of gold, silver, platinum and mercury decomposed to halogen and metal during fine grinding in a mortar but melt or sublime undecomposed when heated. In the publications, the mechanical energy was for the first time pointed out to initiate chemical reactions and locate heating is not the only possible mechanism for initiating chemical reactions by mechanical actions. The effects of mechanical energy can be classified in these aspects:

- Mechanical activation: using mechanical energy to enhance a reaction during subsequent processing.
- Mechanochemistry: using mechanical energy to induce a reaction whilst the energy is being input to the system.
- Mechanical milling: milling of a pure metal or compound that is in a state of thermodynamic equilibrium at the start of milling.
- Mechanical alloying: using mechanical energy to cause solid solution of element during energy input.

Mechanochemistry has been mostly for inorganic solids. However, application of this method to purely organic reactions has not been well developed before the pioneering and systematic work by Toda and coworkers. The solid state reactions that are a branch of solvent free reactions are

attractive not only because of the simplicity of the procedure, but also particularly from the view point of environmental protection because there is no need for the use of harmful organic solvent. Some novel products can be obtained only from solvent-free reactions rather than from the liquid phase reactions. Other advantages of this method are easier workup, shorter reaction time and high yields. Therefore solid state reactions or solvent free reactions are one of the most important synthesis techniques in green chemistry. Binaphthol is a typical chiral compound with strong dissymmetry and can be resolved easily into antipodes in high purity. Binaphthol also has important applications in many businesses such as organic syntheses, dyes, agriculture chemicals, etc., especially in special medicines. Some oxidative coupling reactions of phenols in the presence of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ proceed faster and more efficiently in the solid state than in solution. When a mixture of beta naphthol and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ was finely powdered using an agate mortar and pestle, and then the mixture was kept at 50°C for 2hr, Binaphthol was obtained in 95% yield after decomposition of the reaction mixture with dilute HCl. In contrast, heating a solution of beta naphthol and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 50% aqueous MeOH under reflux for 2hr gave Binaphthol in 60% yield. Recently, a mechanical technique called High Speed Vibration Mill(HSVM) in mechanochemistry context has been gained popularity in organic synthesis. In this work we report the solvent-free oxidation coupling of beta naphthol by $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ using a HSVM reactor that designed by the authors. This reaction takes place quantitatively within a few minutes and the effects of the particle size of reactants, rate of vibration and mill ratio filling parameters are also investigated.